

PATENT ABSTRACTS OF JAPAN.

(11)Publication number : 06-017080
 (43)Date of publication of application : 25.01.1994

(51)Int.CI. C10M169/02
 // (C10M169/02
 C10M101:02
 C10M105:02
 C10M115:08)
 C10N 30:00
 C10N 30:02
 C10N 30:06
 C10N 40:02
 C10N 50:10

(21)Application number : 04-194946

(71)Applicant : SHOWA SHELL SEKIYU KK

(22)Date of filing : 29.06.1992

(72)Inventor : OZAKI KOYO
 TANAKA KEIJI
 TSUCHIYA TETSUO

(54) UREA GREASE COMPOSITION

(57)Abstract:

PURPOSE: To obtain a urea grease which exhibits a desired consistency though it contains a small amt. of a thickener and which is excellent in mechanical stability, water resistance, pumpability, and acoustic characteristics.

CONSTITUTION: A diurea compd. of the formula: R1NHCONHR2NHCONHR3 (wherein R2 is a tolylene group; and R1 and R3 are each a 16-18C linear or branched satd. or unsatd. hydrocarbon group) is mixed with a diurea compd. of the formula: R4NHCONHR5NHCONHR6 (wherein R5 is a diphenylmethane group; and R4 and R6 are each an 8C linear or branched satd. hydrocarbon group) in a molar ratio of the latter compd. to the former of 0.2-0.9, giving a thickener. A urea grease compsn. is prep'd. by mixing a mineral oil and/or a synthetic oil as the base oil with the thickener in an amt. of 2-20wt.%.

LEGAL STATUS

[Date of request for examination] 23.08.1995

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 2864473

[Date of registration] 18.12.1998

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

(57)
[ABSTRACT]

[PURPOSE]

A desired butterfly degree is provided in minor amount of thickener, it is an offer of urea grease superior in stability of winning machine, water resistance, *oshiso* characteristics and acoustic properties.

[CONSTITUTION]

Nitramine, (a) Diurea compound shown with R¹ NHCONHR² NHCONHR³ (tolylene group, R¹ and R³ are alkyl group of linear or carbon number 16-18 or branch-shaped saturation or unsaturation R² out of equation) and (b) urea system grease composition including rate 20-90 which was the mixture that it was than diurea chemical agent shown in with R⁴ NHCONHR⁵ NHCONHR⁶ (diphenyl-methane group, R⁴ and R⁶ are saturation alkyl group of normal chain of carbon number 8 or branch connection R⁵ out of equation) (a) (b) having made contain 2-20 % by weight for the base oil that it was than liquid petrolatum and synthetic oil in the thickener which was mol %.

[WHAT IS CLAIMED IS:]

[Claim 1]

In; Nitramine, (a) The mixture that it is than diurea chemical agent shown with diurea compound shown with R¹ NHCONHR² NHCONHR³ (tolylene group, R¹ and R³ are alkyl group of linear of carbon number 16-18 or branch-shaped saturation or unsaturation R² out of equation) and (b) R⁴ NHCONHR⁵ NHCONHR⁶ (diphenyl-methane group, R⁴ and R⁶ are saturation alkyl group of normal chain of carbon number 8 or branch connection R⁵ out of equation); Urea system grease composition; wherein; (a) Proportion 20-90 which it was similar; and taisuru (b) made contain 2-20 % by weight for the base oil that it was than mineral oil and synthetic oil in the thickener which was mol %.

[DETAILED DESCRIPTION OF THE INVENTION]

[0001]

[INDUSTRIAL APPLICATION FIELD]

The present invention relates to the urea grease composition which annexed two kinds of diurea compound as thickener.

[0002]

[PRIOR ART]

In late years, Progress of machine art is remarkable, and, in various industry such as auto or iron and steel, miniaturization of various machine part, lightweighting and *koseinoka* advance, a lubrication point tends to be in high temperature. Because of this, Because urea system grease superior in heat resistance and oxidation stability is effective in extension of cutbacks and machine life time of the application quantity, the application is opened in gradual. Art of Japanese Patent Laid-Open No. 1-284591 discloses diurea compound as thickener, other, this diurea compound comprises alkyl group of carbon number 16-18 in alkyl group of carbon number three or four in on the other hand of diphenyl-methane radical of 2 values. Art of Japanese Patent Laid-Open No. 1-268793 employs diurea compound as thickener, too, but, radical chosen by the group that it was than octadecyl group, octyl radical, dodecyl group coupled this diurea compound with both ends of diphenyl-methane radical of 2 values and mole fraction 20-80 of the octadecyl group were done with mol %. Japanese Patent Laid-Open No. 1-139696 relate to technology using diurea chemical agent of type having urea

bond at both ends of diurea chemical agent of type having urea bond as thickener at both ends of diphenyl-methane and tolylene radical or *bitoriren* radical together, but, the radical which was coupled with tolylene radical or *bitoriren* radical the alkyl which urea bond was gone through, and was coupled with diphenyl-methane radical was limited to a thing of carbon number 8 and urea bond was gone through was limited to aromatic group. Japanese Patent Laid-Open No. 2-77494 relate to technology using diurea chemical agent of type having urea bond at both ends of diurea chemical agent of type having urea bond as thickener at both ends of diphenyl-methane and *bitoriren* radical together. Art of Japanese Patent Publication No. 63-26798 uses three kinds of diurea compound together, but, radical both the same radical caught in two urea bond is employed, both ends put a thing of octyl together, and chemical agent of one already employs a thing of octyl in dodecyl end thing of dodecyl, chemical agent 1 of one radical of both ends chemical agent of one another end. Art of Japanese Patent Laid-Open No. 58-185693 dopes alkenyl amber acid amide, alkylbenzene sulfonic acid metal salt, petroleum sulfonic acid metal salt in diurea system grease, and acoustic properties are improved, but, there is not trait about diurea particularly.

[0003]

[PURPOSE]

There is first object of the present invention in point a desired butterfly degree is provided in minor amount of thickener and provide superior urea grease in stability of machine, water resistance, *oshiso* characteristics and acoustic properties. Secondary purpose of the present invention expands art of Japanese Patent Laid-Open No. 1-139696, there is at a point to improve.

[0004]

[CONSTITUTION]

Urea system grease composition including rate 20-90 which is the mixture that it is than the diurea chemical agent which the present invention is shown in with diurea compound shown with nitramine (a) $R^1 \text{NHCONHR}^2 \text{NHCONHR}^3$ (tolylene group, R^1 and R^3 are alkyl group of linear of carbon number 16-18 or branch-shaped saturation or unsaturation R^2 out of equation) and (b) $R^4 \text{NHCONHR}^5 \text{NHCONHR}^6$ (diphenyl-methane group, R^4 and R^6 are saturation alkyl group of normal chain of carbon number 8 or branch connection R^5 out of equation) (a) (b) having made contain 2-20 % by weight for the base oil that it is than liquid petrolatum and synthetic oil in the thickener which is mol % is related to.

[0005]

(a) Component can be got by reacting octadecylamine. Tolylene diisocyanate 2,4- can employ tolylene diisocyanate, 2,6- tolylene diisocyanate and these mixtures. (b) Component is provided by reacting diphenyl-methane diisocyanate and octyl amine.

[0006]

[EXAMPLE]

As follows, Embodiment is advocated, and the present invention is explained. In addition, Comparative examples are written jointly, character of urea grease of the present invention was clarified. Begin grease kettle, and base oil of diisocyanate and 60 part by weight of component (a) is heated to about 80 degrees Celsius in blending ratio of coal shown in example 1 to table 7, after having dissolved diisocyanate, it is drastic, and n- octadecylamine of the constituent which made this dissolve in base oil of 20 part by weight (a) is stirred as well as gradual. After, about 10 minutes later, having added 4,4 diphenyl-methane diisocyanate of component (b), the n- octyl amine which made base oil of 20 ni part by weight dissolve is added, and stirring is continued. Temperature rises by reaction with diisocyanate and amine, but, it makes, in this state, 170 degrees Celsius are heated to after stirring, and conclude reaction for about 30 minutes. Afterwards, *ho* cold water was kneaded to room temperature, and grease was made. The poly alpha - olefin oil that viscosity of mineral oil shown in embodiment is 11cst (100 degrees Celsius) is 12cst (100 degrees Celsius). Resultant of sound examination was shown in table in butterfly degree (25 degrees Celsius, non-misce) range after a butterfly degree, drip-point of grease of each embodiment, shell roll (150 degrees Celsius, 24h) and heat in 180 degrees Celsius. In addition, The sound check measured by

method as claimed in Japanese Patent Publication No. 53-2357. Begin grease kettle, and 80 part by weight of diisocyanate and mineral oil is heated to about 80 degrees Celsius in blending ratio of coal shown in comparative example one or two, after having dissolved diisocyanate, the amine which made dissolve in liquid petrolatum of 20 part by weight was added, and it was stirred. After, in this state, having continued a churn for about 30 minutes, it makes about 170 degrees Celsius are heated to, and reaction conclude, it was kneaded after *horei* in room temperature, and grease was made. Attribute of each comparative example was shown in table. (following blank) [0007]

[TABLE 1]

		実施例 1	実施例 2	実施例 3	実施例 4	実施例 5
成 分 (a)	(1) 2,4/2,6 (80%/20%)— トリレンジイソシアネート g (2) n-オクタデシルアミン g	3.22 9.58	2.82 8.38	2.02 5.98	1.21 3.59	0.40 1.20
成 分 (b)	(1) ジフェニルメタン4,4'- ジイソシアネート g (2) n-オクチルアミン C 8 g	1.57 1.63	2.36 2.44	3.94 4.06	5.51 5.69	7.08 7.32
鉛 油 ボリα-オレフィン 増ちよう剤含有量 (a)/(b) (性 状)	g g % モル% / モル%	184 — 8.0 80/20	184 — 8.0 70/30	184 — 8.0 50/50	184 — 8.0 30/70	184 — 8.0 10/90
ちよう度 (25°C、混和) シェルロール (室温、24時間)	(25°C、混和) g	268 358	255 347	240 317	245 298	271 332
含水1.0%シェルロール (室温、24時間)	g	362	356	347	309	351
シェルロール (150°C、24時間) 音響試験 (120秒後)	g	382 38	369 20	310 25	305 15	321 26

[0008]

[TABLE 2]

		実施例 6	実施例 7	比較例 1	比較例 2
成分 (a)	(1)2.4/2.6 (80% / 20%) + トリレジソシアネートg (2)n-オクタデシルアミンg	0.91 2.69	1.21 3.59	* 7.56 22.44	- -
成分 (b)	(1)ジフェニルメタン4,4'- ジイソシアネート g (2)n-オクチルアミンC8g	4.13 4.27	5.51 5.69	- -	7.87 8.13
鉱油 ポリα-オレフイン 増ちょう剤含有量 (a)/(b) (性状) ちょう度 シェルロール (室温, 24時間)	188 - 6.0 30/70 (25°C, 混和) 292 357	- 184 8.0 30/70 251 313	170 - 15.0 100/0 843 368	184 - 8.0 0/100 342 402	
含水10%シェルロール (室温, 24時間)	382	316	331	412	
シェルロール (150°C, 24時間) 音響試験 (120秒後)	362 20	314 18	>440 47	365 51	

* 2.4/2.6 ratios in a comparative example are 65% /35%. A butterfly degree: JIS2220 shell roll: It is conformity sound check in ASTMD1831: Method as claimed in Japanese Patent Publication No. 53-2357 is depended on.

[0009]

The grease which added oxidation inhibitor, anti-rust agent and extreme pressure agent to embodiment 6 for embodiment 8, 3-5 comparative example embodiment 8 is made, the resultant which compared commercial grease and attribute was shown in the next table. Mechanical soundness, water resistance, heat resistance and *oshiso* are sexual, and present invention grease is more superior than commercial grease.

[TABLE 3]

	実施例 8	比較例 3	比較例 4	比較例 5
(性 状) ちよう度 (25°C、混和) シェルロール (室温、24時間)	313 360	272 414	320 365	338 >440
含水10%シェルロール (室温、24時間)	381	>440	>440	>440
シェルロール (150°C、24時間) 見掛粘度 0°C. 10 sec ⁻¹ (ボアズ)	372 910	>440 —	>440 1180	>440 1800

(note) Comparative example 3:00 Commercial urea system grease comparative example 4:00 Commercial lithium soap base grease comparative example 5:00 Grease pro-calcium inferiority complex [0010]

Example 1 to 4 of 6-10 comparative example Japanese Patent Laid-Open No. 1-139696 and nine data are shown for comparative example 6-10.

[TABLE 4]

	比較例 6	7	8	9	10
特開平1-139696号 発明の対応実施例番号	1	2	3	4	9
成 分 (a)	(1)ジフェニルメタン4,4'-ジイソシアネート g (2)オクチルアミンC8 g	8.98 9.29	6.08 6.29	3.09 3.19	6.69 6.92
成 分 (b)	(3)3,3'-ビトリレン-4,4'-ジイソシアネート g (4)2,4/2,6 (65%/35%) -トリレンジイソシアネートg (5)パラトルレイシン g	3.16	6.42	9.79	8.16
鉛 油 g ポリα-オレフィン g 増ちよう剤含有量 % (a)/(b) モル% / モル%	176 8 12 75/25	176 12 12 50/50	176 12 12 25/75	176 12 12 50/50	176 12 50/50
性 状 ちよう度 (25°C、混和) シェルロール (150°C、24時間)	270 283	285 296	295 325	290 341	283 291
150°C加熱後ちよう度 (25°C、不混和)	178	175	174	177	179
音響試験 (1.20秒後)	52	59	67	48	49

Judging from this table, it will be clear that the present invention compares with devise of Japanese Patent Laid-Open No. 1-139696, and acoustics is improved more generally. More according to the current invention, hard grease is provided with quantity of a little thickener. If it is got whether it is good, it is obvious that butterfly degree yield is good. As thus described urea system grease composition of the present invention is superior in butterfly degree yield as well as betterment of acoustic properties , besides, superior grease can be provided in stability of machine in high temperature from water resistance and room température.

[0011]

[EFFECT]

The present invention made art richness by showing blending of new thickener, and the acoustic properties which were the main problem of the Japanese Patent Laid-Open No. 1-139696 that was anticipation were able to be improved still more. Besides, Grease of the present invention is superior in mechanical soundness in high temperature, there is a very little thermosetting property.